

**DATA VALIDATION REPORT**  
**MICROBAC LABORATORY SDG L13101612**

Project / Site Name: Environmental Remediation Services at White Sands Missile Range (WSMR), NM; CCWS-11, Open Burn/Open Detonation (OB/OD) Area

Project No.: 139791

Data Reviewer: M. Lyon, Shaw Environmental, Inc. a CB&I Company

Review Date: December 4, 2013

Matrix: Groundwater, 2 field samples plus extra volume for MS/MSD

Parameters: Perchlorate 6850  
Explosives 8330B  
Nitrate + Nitrite, as Nitrogen 353.2

Validation Level: EPA Level III

Laboratory: Microbac Laboratories, Inc. Ohio Valley Division

Sample Delivery Group L13101612

Sample Nos.: HTA17-1013-1 and HTA3-1013-1.

Comments: Extra volume submitted with HTA3-1013-1 for MS/MSD in this SDG.

The data were reviewed and qualified according to the *Sampling and Analysis Plan/Quality Assurance Project Plan, Environmental Remediation Services, White Sands Missile Range, New Mexico October 2010; Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 4.2, 2010*; laboratory-specific statistical process control criteria, and the analytical method specific requirements.

## DATA VALIDATION REQUIREMENTS

Level IV or Full Validation includes all parameters listed below. Level III Cursory Validation parameters are indicated by an asterisk (\*).

### Organic Parameters

- \* Temperature
- \* Holding times
- GC/MS instrument performance check
- \* Initial and continuing calibrations
- \* Blanks
- \* Surrogate recoveries
- \* Matrix spike/matrix spike duplicate
- \* Laboratory control sample / blank spike
- \* Field duplicate
- \* Internal standard performance
- Target compound identification
- Tentatively identified compounds
- Compound quantitation
- Reported detection limits
- System performance
- \* Overall data assessment

### Inorganic and General Chemistry Parameters

- \* Temperature
- \* Holding times
- \* Initial and continuing calibration
- \* Blanks
- \* Matrix spike/matrix spike duplicate
- \* Laboratory control sample / blank spike
- \* Field duplicate
- \* Matrix duplicate
- ICP interference check sample
- CVAA / GFAA quality controls
- \* ICP serial dilution
- Sample results verification
- Analyte quantitation
- Reported detection limits
- \* Overall data assessment

## DATA VALIDATION QUALIFIER DEFINITIONS

No qualifier indicates that the data are acceptable both qualitatively and quantitatively.

- U Not detected. The analyte was analyzed for but was not detected above the level of the associated value. The associated value is the Limit of Quantitation (LOQ).
- J Estimated. The analyte was detected and positively identified. The associated numerical value is the approximate concentration of the analyte in the sample and the bias is in determinable.
- J- Estimated. The analyte was detected and positively identified. The associated numerical value is the approximate concentration of the analyte in the sample and the bias is determined low due to associated quality control indicators.
- J+ Estimated. The analyte was detected and positively identified. The associated numerical value is the approximate concentration of the analyte in the sample and the bias is determined high due to associated quality control indicators.
- N Tentatively identified. The analysis indicates the presence of an analyte for which there is presumptive evidence to make a tentative identification.
- UN Tentatively not detected, the LOQ is estimated. The analyte was analyzed for but was not detected above the reported LOQ. However, the reported LOQ is an estimate and may not be accurate or precise.
- NJ Tentatively identified. The reported concentration is an estimate. The analysis indicates the presence of an analyte for which there is presumptive evidence to make a tentative identification and the associated numerical value represents the approximate concentration.
- R Rejected. The data are not usable. The presence or absence of the analyte cannot be confirmed.

## DATA VALIDATION QUALIFIER REASON CODES

Reason Code	Data Quality Condition Resulting in Assigned Qualification
<b>General Use</b>	
FB	Field blank contamination
FD	Field duplicate evaluation criteria not met
HT	Holding time requirement was not met
PR	Preservation requirements not met
LCS	Laboratory control sample evaluation criteria not met
MB	Method blank or preparation blank contamination
RB	Rinsate blank contamination
TB	Trip blank contamination
SDL	Sample quantitation limit exceeds decision criteria and the analyte was not detected
<b>Inorganic Methods</b>	
CCB	Continuing calibration blank contamination
CCV	Continuing calibration verification evaluation criteria not met
D	Laboratory duplicate precision evaluation criteria not met
DL	Serial dilution results did not meet evaluation criteria
ICS	Interference check sample evaluation criteria not met
ICV	Initial calibration verification evaluation criteria not met
MS	Matrix spike recovery outside acceptance range
PDS	Post-digestion spike recovery outside acceptance range
MSA	Method of standard additions correlation coefficient < 0.995
PB	Preparation blank
<b>Organic Methods</b>	
CCAL	Continuing calibration evaluation criteria not met
ICAL	Initial calibration evaluation criteria not met
ID	Target compound identification criteria not met
IS	Internal standard evaluation criteria not met
MS/MSD	Matrix spike/matrix spike duplicate accuracy and/or precision criteria not met
SUR	Surrogate recovery outside acceptance range
TUNE	Instrument performance (tuning) criteria not met
P	The detected concentration difference between the primary and secondary column is greater than 25%.

**SAMPLE DELIVERY GROUP L13101612**  
**LEVEL III DATA VALIDATION SUMMARY**

Analysis / Method	Temperature	Holding Times	Calibration	Blanks	Surrogate	MS/MSD	LCS	Duplicate	Other
Perchlorate 6850	✓	✓	✓	✓	NA	NA	✓	NA	NA
Explosives 8330B	✓	✓	✓	✓	✓	✓	✓	✓	✓
Nitrate+Nitrite-N 353.2	✓	✓	✓	✓	NA	✓	✓	✓	✓

**Notes:**

✓ Indicates that all quality control criteria were met for the parameter(s)

N/A Indicates the validation criteria is not applicable to the analysis

If validation criteria were not met and the data were qualified, then details can be found at the page number indicated in the table.

**DATA ASSESSMENT**  
**PERCHLORATE (Method 6850)**

**I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

**II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

**III. Calibration**

A. Calibration coefficient of determination, alternate source calibration verification, and CCV, were reviewed and found compliant.

**IV. Blanks**

A. Method blank and CCB analysis results were reviewed and found to be in compliance.

**V. MS/MSD**

A. MS/MSD analyses were performed on sample HTA3-1013-1. The parent sample concentration exceeded the spike levels by more than 4-times making the MS/MSD meaningless. MS/MSD could not be evaluated.

**VI. LCS**

A. The LCS results were reviewed and found to be in compliance.

**VII. Duplicate**

A. MSD was the only precision result reported. MSD precision measurement was not meaningful.

**VIII. Other**

A. The sample HTA17-1013-1 was analyzed at 10,000-times dilutions due to high concentrations of the target analyte.

## **EXPLOSIVES (Method 8330B)**

### **I. Temperature**

- A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

- A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

- A. Calibration coefficient of determination, alternate source calibration verification, and CCV were reviewed and found compliant with exceptions. Percent difference recoveries for the compound tetryl failed criteria in alternate source calibration check on the instrument designated HPLC4. The applicable analysis results from instrument HPLC4 were the confirmation and 5-times dilution analyses for sample HTA17-1013-1. Tetryl results from those analyses are qualified estimated non-detect with “UN.” Reportable analysis results were taken from instrument HPLC5 which had compliant calibration checks.

### **IV. Blanks**

- A. Method blank and CCB analysis results were reviewed and found to be in compliance.

### **V. Surrogate Spikes**

- A. Surrogate spike recoveries were compliant in all sample analyses.

### **VI. MS/MSD**

- A. MS/MSD analyses were performed on sample HTA3-1013-1. Bias and precision results met acceptance criteria.

### **VII. LCS**

- A. The LCS results were reviewed and found to be in compliance.

### **VIII. Duplicate**

- A. MSD precision results met acceptance criteria.

### **IX. Other**

- A. Valid confirmation analysis results were reported for all analyte detections.

## **NITRATE + NITRITE AS NITROGEN (Method 353.2)**

### **I. Temperature**

- A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

- A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

- A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

- A. Method blank was reviewed and found to be in compliance.

### **V. MS/MSD**

- A. MS/MSD analyses were performed on sample HTA3-1013-1. The MSD percent recovery exceeded the upper acceptance limit while the MS recovery was compliant. Validation qualifiers were not assigned..

### **VI. LCS**

- A. The LCS results were reviewed and found to be in compliance

### **VII. Duplicate**

- A. The MSD precision result met acceptance criteria. The laboratory also analyzed a LCS duplicate. The results were reviewed and found to be compliant.

### **VIII. Other**

- A. LOQ was reviewed and found compliant.



## **DATA QUALIFICATION SUMMARY**

**CCWS-11, Open Burn/Open Detonation (OB/OD) Area, two samples; HTA17-1013-1 and HTA3-1013-1.**

### **Perchlorate – Data Qualification Summary**

No sample data were qualified in this SDG.

### **Explosives – Data Qualification Summary**

No reportable sample data were qualified in this SDG. A low recovery for tetryl in an alternate source check (initial calibration check) was noted and the tetryl result qualified “UN” for non-detect and estimated in the confirmation and dilution analyses which were not reportable.

### **Nitrate plus nitrite – Data Qualification Summary**

No sample data were qualified in this SDG.

## **OVERALL ASSESSMENT OF DATA**

### **I. Compliance with method and project requirements**

- A. All analyses were performed within the analytical methods specifications and project requirements.

### **II. Usability**

- A. Based on the quality control criteria reviewed, all unqualified data are usable for project purposes. No reportable data results were qualified during data validation. No data results were rejected as unusable. Data qualifiers assigned by the laboratory in the analytical report include the “J” qualifier when analytes were identified but at concentrations less than the LOQ. Estimated results are usable for limited purposes.